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THE EVALUATION OF AMBIENT-TEMPERATURE PROCESSES FOR REPAIR BONDING OF ALUMINUM ALLOYS

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On-aircraft bonded repair processes typically require impractical surface preparation processes and controlled elevated-temperature heating in order to attain desired performance. Surface preparations are critical for bonded joint strength and durability but tend to be difficult, time-consuming, and contain hazardous materials. Elevated temperatures may be required to cure adhesives, dry the repair area, dry silane coupling agents, and/or cure bond primer. Due to time constraints and/or the inability to properly heat the repair area, aircraft maintainers often require a low-temperature bonding process that is quick and easy to conduct. In this study, epoxy paste adhesives were evaluated using three surface preparations in order to identify a practical, high-performance, low-temperature bonded repair process. Aluminum adherends prepared using a nonhazardous nylon-pad/sol-gel process were bonded with paste adhesives. Mechanical strength and durability results were compared to both the industry standard surface preparation for aluminum (phosphoric acid anodization with Cytec BR 127 bond primer) and a commonly used scuff-sand/solvent wipe process. The nylon-pad/sol-gel process outperformed scuff-sand/solvent wipe in all cases and, in some instances, approached the performance of phosphoric acid anodization. Scuff-sand/solvent wipe led to reduced initial strength in many instances and moisture durability in all cases.

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PREFACE

This report covers work performed by the University of Dayton Research Institute (UDRI), Dayton, Ohio 45469-0138, during the period from January 2000 to December 2001. All work was performed under Air Force Contract F33615-95-D-5616, Delivery Order 0007 and F33615-00-D-5600, Delivery Order 0001. The work was administered under the direction of the Systems Support Division of the Air Force Materials and Manufacturing Directorate, Air Force Research Laboratory, Wright-Patterson Air Force Base, Ohio. Lt. Heather Crooks was the Contract Monitor. Mr. James J. Mazza was the government engineer and technical leader. The UDRI Program Manager was Robert Askins. The Principal Investigator was Mr. Daniel McCray. Mr. James Huff and Mr. Jeffrey Smith, UDRI, performed specimen fabrication and testing associated with this project. Appreciation is extended to Kylie Huber and Carly Wreesman, Southwestern Ohio Council for Higher Education, for laboratory technical support.

The author acknowledges the Strategic Environmental Research and Development Program (SERDP) for partially funding this effort as well as other related sol-gel research. Advice and technical input provided by Jay Fiebig and Bill Schweinberg of Warner Robins Air Logistics Center (WR-ALC/TIEDD) are greatly appreciated. Thanks are also extended to Dr. Kay Blohowiak, Boeing Phantom Works, for development of the sol-gel chemistry used in this program.

1 BACKGROUND

On-aircraft adhesive bonding of metal substrates typically requires the use of impractical surface preparation processes in order to attain the desired bonded joint strength and durability. Surface preparations currently used for on-aircraft repairs rely on hazardous materials and/or inconvenient processing steps, or they do not yield adequate bond performance. Many U.S. Air Force Technical Orders (T.O.s) require the use of surface preparations such as phosphoric acid anodize (PAA) or acid paste etches for the repair of aluminum alloy structure. These surface preparations are often impractical due to difficulties imposed by the on-aircraft use of acids or the extended time required for their application. The phosphoric acid in PAA and sulfuric acid used in common paste acid etches are difficult to contain and can embrittle certain high-strength steels[1]. In addition, the acids must be rinsed after application and could cause corrosion of metallic structure if not completely removed. The grit-blast/silane surface preparation employed in many repair applications provides an alternative to the use of acids but requires a grit-blasting step, elevated-temperature drying, and several hours to perform[2]. The use of hazardous materials in these surface preparations is becoming more difficult due to existing and proposed environmental and health regulations. The combination of these regulations and the need to reduce environmental waste stream while maintaining adequate bond performance has lead to the development of more environmentally friendly processes.

The need for controlled elevated-temperature heating further complicates on-aircraft bonded repair processes. Heating may be required to cure adhesives as well as dry substructure, dry silane coupling agents and/or cure bond primer. Film adhesives are often chosen for aircraft repair applications due to the better overall mechanical properties, lower porosity, and more uniform bondlines obtainable with their use when compared to similar joints prepared using paste adhesives. However, most repair film adhesives must be cured at temperatures of 180°F or higher.

Some current US Air Force maintainers utilize easier processes for on-aircraft repair despite reductions in mechanical strength and/or durability. These include the use of scuff-sand/solvent

wipe surface preparations and/or ambient-temperature-curing, two-part paste adhesives. These processes are performed due to the desire or necessity to conduct repair bonding as quickly as possible and without the use of elevated-temperature heating. Maintenance personnel across the Air Force have become accustomed to scuff-sand/solvent wipe as a surface preparation to meet the time limitations placed on them by mission requirements and operations tempo. However, maintenance personnel are not always authorized to perform bonded repairs on USAF aircraft using the scuff-sand/solvent wipe process or two-part epoxy paste adhesive materials.

Currently, a need exists for an ambient-temperature adhesive bonding process including a practical, high-performance on-aircraft surface preparation process. Several US Air Force maintenance personnel identified several two-part paste adhesives they wanted to see evaluated. The University of Dayton of Research Institute (UDRI) performed screening tests on these adhesives under U.S. Air Force contract F33615-95-D-5616, Delivery Order 001. UDRI evaluated an ambient-temperature on-aircraft repair process for aluminum utilizing a nylon-pad/sol-gel process that has demonstrated feasibility as a prebond surface preparation for aluminum[3]. The sol-gel surface preparation was used without primer in this evaluation so the entire bonding process could be conducted at ambient temperature and without the use of hexavalent chromium. Nylon-pad/sol-gel test results were compared to those obtained using bonded specimens prepared by the PAA/BR 127 and scuff-sand/solvent wipe processes.

2 EXPERIMENTAL

This evaluation of ambient-temperature adhesive bonding processes included a determination of initial paste adhesive strengths as well as an investigation into the effect of surface preparation on bond strength and moisture durability. The two-part epoxy paste adhesives evaluated in the program included Hysol EA 9309.3NA, EA 9320NA, EA 9330.3, EA 9394, and EA 9396 from Henkel Loctite as well as 3M Company EC 2615 and EC 3333. Initial strengths were determined via tensile lap shear (ASTM D 1002)[4] and floating roller peel testing (ASTM D 3167)[5], using Al 2024-T3 adherends prepared with PAA[6] and Cytec BR 127 adhesive bond primer. Wedge tests (ASTM D 3762)[7] were also conducted on Al 2024-T3 prepared with PAA/BR 127 and bonded with the evaluated paste adhesives. Polyester random mat scrim cloth

(0.004 in thick) was used for bondline control with all adhesives except EA 9309.3NA which is manufactured with glass beads to control bondline thickness to approximately 0.005 inch. All data represent the average of five specimens unless otherwise noted.

Surface preparation prior to bonding is the key for achieving long-term moisture durability. Data were generated comparing three surface preparations: a scuff-sand/solvent wipe process (often used for on-aircraft bonding), a relatively new nylon-pad/sol-gel surface preparation, and PAA/BR 127 as a control process. The nylon-pad/sol-gel surface preparation was based on The Boeing Company's sol-gel chemistry[8] and in order to produce a simple, ambient-temperature process, did not include a bond primer. Tensile lap shear, floating roller peel, and wedge crack extension tests were used to evaluate the strength and moisture durability of adhesive bonds with the sol-gel surface preparation on Al 2024-T3 adherends. Results obtained were compared to those generated using paste adhesives with the PAA /BR 127 and scuff-sand/solvent wipe processes. All data represent the average of five specimens unless otherwise noted.

Finally, a repeatability assessment was conducted using the adhesive that exhibited the best results from the initial screening. Lap shear and wedge tests were conducted using Al 7075-T6 adherends prepared using PAA/BR 127, nylon-pad/sol-gel, and grit-blast/sol-gel surface preparations. Both sol-gel processes were based on the Boeing chemistry and used without bond primer. A total of five panels (25 specimens) per condition were fabricated.

2.1 Determination of Baseline Adhesive Properties

Evaluation of baseline properties of the various paste adhesives was conducted on phosphoric acid anodized Al 2024-T3 primed with Cytec BR 127 bond primer and tested via tensile lap shear and floating roller peel. BR 127 was applied according to Cytec's recommended procedure to a nominal dry film thickness of 0.0002 inch, dried for 30 minutes at ambient temperature (70°F), and then cured for 60 minutes at 250°F. Adherends were bonded with the epoxy paste adhesives according to manufacturers' recommendations and cured at ambient temperature (70°F $\pm$ 5°F) using either 35 psi positive pressure or 15 in Hg vacuum pressure. Vacuum pressure was applied in order to replicate on-aircraft curing conditions. Pressure was

applied to the panels for only the first 24 hours. Panels were then held at 70°F for an additional 6 days.

Elevated-temperature curing of paste adhesives is a common repair practice necessitated by the desire to decrease the amount of time required to perform repairs. For this reason, additional lap shear and peel specimens were fabricated using adhesive cured at elevated temperatures recommended by the manufacturers. These temperatures were well below the cure temperatures required for most epoxy film adhesives. Panels bonded at elevated temperature were heated at a rate of 5°F per minute to the recommended cure temperature and held at that temperature for 60 minutes. The recommended cure temperature was 180°F for all adhesives except EA 9394 and EA 9396. The cure temperature for these adhesives was 150°F. Tensile lap shear specimens were tested at 70°F, 160°F, and 180°F. Floating roller peel testing was conducted at 70°F. Failure modes were determined and recorded as percent cohesive failure (within the adhesive layer). Specimens prepared with PAA/BR 127 exhibited varying percentages of interfacial failure between the primer and adhesive but did not fail at the aluminum-primer interface. Results of mechanical testing performed on specimens bonded with adhesives cured at ambient temperature (70°F) under positive pressure are shown in Table 1. Table 2 contains the results of mechanical tests for specimens bonded with adhesives cured at elevated temperature under positive pressure. Lap shear and peel test results obtained with adhesives cured at elevated temperature under vacuum pressure are shown in Table 3.

Table 1: Comparison of Paste Adhesive Properties when Cured at Ambient Temperature Under Positive Pressure

Adhesive	Lap Shear Strength (psi) (% cohesive failure)			Peel Strength (pli)
	70°F	160°F	180°F	70°F
EA 9309.3NA	4037 (97% co)	754 (94% co)	436 (99% co)	55.9 (100% co)
EA 9320NA	4620 (99% co)	1126 (100% co)	843 (100% co)	23.9 (100% co)
EA 9330.3	4414 (100% co)	764 (100% co)	582 (98% co)	38.4 (100% co)
EA 9394	4076 (94% co)	2522 (100% co)	2697 (100% co)	25.6 (97% co)
EA 9396	5248 (98% co)	3288 (100% co)	2993 (91% co)	24.5 (100% co)
EC 2615	4870 (11% co)	801 (90% co)	714 (39% co)	77.0 (90% co)
EC 3333	3477 (-0-% co)	1087 (-0-% co)	453 (27% co)	75.9 (100% co)

Note: all data are the average of 5 specimens except for EA 9309.3NA (10 specimens)

Table 2: Comparison of Paste Adhesive Properties when Cured at Elevated Temperature Under 35 PSI Positive Pressure

Adhesive	Lap Shear Strength (psi) (% cohesive failure)			Peel Strength (pli)
	70°F	160°F	180°F	70°F
EA 9309.3NA	4872 (40% co)	3142 (90% co)	1274 (60% co)	60.0 (90% co)
EA 9320NA	5756 (99% co)	3597 (81% co)	1708 (84% co)	25.2 (100% co)
EA 9330.3	5344 (93% co)	1022 (86% co)	773 (64% co)	47.6 (100% co)
EA 9394	4857 (78% co)	3126 (84% co)	3141 (84% co)	25.8 (81% co)
EA 9396	4238 (40% co)	4456 (94% co)	3965 (53% co)	17.6 (95% co)
EC 2615	5167 (11% co)	3253 (30% co)	2023 (24% co)	43.6 (3% co)
EC 3333	5177 (2% co)	2919 (32% co)	1690 (14% co)	46.8 (5% co)

Note: all data are the average of 5 specimens except for EA 9309.3NA (10 specimens)

Table 3: Comparison of Paste Adhesive Properties when Cured at Elevated Temperature Under 15 Inches of Hg Vacuum Pressure

Adhesive	Lap Shear Strength (psi) (% cohesive failure)			Peel Strength (pli)
	70°F	160°F	180°F	70°F
EA 9309.3NA	4460 (90% co)	2506 (100% co)	736 (80% co)	52.1 (90% co)
EA 9320NA	5197 (98% co)	3147 (100% co)	1812 (97% co)	27.3 (100% co)
EA 9330.3	5000 (93% co)	729 (88% co)	662 (81% co)	48.1 (100% co)
EA 9394	3818 (93% co)	2926 (95% co)	2798 (98% co)	23.2 (89% co)
EA 9396	3766 (62% co)	3021 (89% co)	2161 (38% co)	13.9 (95% co)
EC 2615	5090 (35% co)	2293 (59% co)	1776 (33% co)	52.5 (5% co)
EC 3333	5225 (13% co)	3280 (36% co)	1810 (13% co)	49.9 (-0-% co)

Note: all data are the average of 5 specimens except for EA 9309.3NA (10 specimens)

Baseline wedge testing was performed on Al 2024-T3 adherends prepared with PAA/BR 127. The adhesives were cured for 60 minutes at 180°F and 35 psi except for EA 9394 and EA 9396 which were cured for 60 minutes at 150°F and 35 psi. Polyester scrim cloth was used for bondline control with all adhesives except EA 9309.3 since it is manufactured with glass beads. Specimens were tested at 120°F and 95-100% relative humidity (RH). Results of the baseline wedge testing are shown in Table 4.

Failure modes for specimens fabricated with all of the Hysol adhesives were mostly cohesive (within the adhesive) with small amounts of interfacial failure occurring between the primer and adhesive. However, the 3M Company adhesives exhibited large amounts of interfacial failure between the primer and adhesive. This failure mode was verified by energy-dispersive spectrometry (EDS) analysis. The 3M adhesives exhibited high peel strengths when compared to

the Hysol adhesives but the poor wedge test results and low percentages of cohesive failure are a concern.

Table 4: Baseline Extension Test Results

Adhesive	Initial (in)	Cummulative Crack Growth (in)							Total (in)	Failure Mode
		1 hr	8 hrs	24 hrs	7 days	14 days	21 days	28 days		
EA 9309.3NA	1.42	0.06	0.10	0.15	0.36	0.43	0.48	0.58	2.00	88% co
	1.33	0.04	0.06	0.10	0.26	0.37	0.50	0.51	1.84	100% co
EA 9320NA	1.59	0.06	0.06	0.07	0.09	0.12	0.15	0.17	1.76	100% co
EA 9330.3	1.38	0.08	0.47	0.73	0.96	1.02	1.06	1.10	2.48	71% co
EA 9394	1.75	0.07	0.16	0.16	0.20	0.22	0.22	0.23	1.98	96% co
EA 9396	1.77	0.07	0.16	0.16	0.29	0.33	0.39	0.42	2.19	100% co
EC 2615	1.33	0.01	0.06	0.14	0.37	0.52	0.60	0.69	2.02	10% co
EC 3333	1.29	0.03	0.18	0.27	0.54	0.67	0.76	0.86	2.15	22% co

The adhesives exhibited a range of mechanical properties. In general, the following trend is observed: as the peel strength of the adhesive increases, the resistance to heat is reduced as exhibited by the reduction in lap shear strength at elevated temperature. It is also seen that curing under vacuum pressure decreases the strength of the bonds. This is likely due to the increased porosity observed in the bondline for vacuum-cured specimens[9]. Finally, a reduction in strength at elevated temperature is detected when curing at ambient temperature compared to an elevated-temperature cure. This is likely caused by reduced glass transition temperature (T_g)[10] as a result of the ambient-temperature cure cycle[11]. Overall, the Hysol adhesives exhibited strengths consistent with published data provided by Hysol and exhibited cohesive failure modes whereas the 3M Company adhesives yielded excessive amounts of interfacial failure mode.

2.2 Surface Preparation Evaluation

Aircraft aluminum bonded parts, as received from the manufacturer, are typically fabricated utilizing high-performance surface preparations such as acid anodization or elevated-temperature acid etch. Variants of these processes are often specified in Air Force T.O.s as the preferred methods for preparing surfaces for adhesive bonded repair. However, due to the difficulty of performing these surface preparations on aircraft, maintainers across the Air Force desire a convenient ambient-temperature adhesive bonding process capable of delivering acceptable strength and durability. Therefore, the paste adhesives evaluated in this program were tested with three different surface preparations. PAA/BR 127 was used as a standard. A scuff-

sand/solvent wipe process was used to replicate a common on-aircraft approach. Finally, an ambient-temperature nylon-pad/sol-gel surface preparation was evaluated.

Bare Al 2024-T3 adherends were used to fabricate all bonded specimens in the surface preparation evaluation. Tensile lap shear, floating roller peel, and wedge tests were conducted to determine initial bond strength and bond moisture durability using the adhesives with each of the three surface preparations. The scuff-sand/solvent wipe process consisted of degreasing by wiping the adherends with lint-free cloths moistened with acetone, abrading with 100-grit Al_2O_3 abrasive paper using a random orbital sander, and final wiping with acetone to remove residue. The nylon-pad/sol-gel process employed the same acetone degrease step. Adherends were then abraded with 3-inch diameter 3M Company Scotch-Brite™ Roloc™ coarse pads using a 20,000-RPM nitrogen-driven rotary grinder. Adherends were not cleaned with solvent after the final abrasion step. Residue was removed using compressed dry, clean, nitrogen. Sol-gel solution was brush-applied keeping the surface wet for 3 minutes and then dried at ambient laboratory conditions for 30 minutes prior to adhesive application.

For all panels, adhesive was mixed according to the manufacturers' recommendations and applied to both bonding surfaces. Polyester random mat scrim cloth was used for bondline control with all adhesives except EA 9309.3NA, since it is manufactured with glass beads for bondline control. All specimens were cured under 15 inches of Hg vacuum pressure in order to replicate on-aircraft curing conditions unless otherwise noted. Scuff-sand/solvent wipe, nylon-pad/sol-gel, and PAA/BR 127 specimens were heated to elevated temperature to accelerate adhesive cure. A second set of nylon-pad/sol-gel specimens was fabricated with adhesive cured at ambient temperature. These data were compared to controls fabricated using the PAA/BR 127 surface preparation with adhesive cure at ambient temperature under 35 psi positive pressure. The elevated-temperature cure for EA 9394 and EA 9396 was 60 minutes at 150°F. The elevated-temperature cure for the other adhesives was 60 minutes at 180°F. The ambient-temperature cure cycle for all the adhesives was 24 hours at 70°F under pressure followed by an additional 6 days at ambient after removal of pressure. Tensile lap shear specimens were tested at 70°F, 160°F, and 180°F. Floating roller peel testing was conducted at 70°F. Wedge testing was conducted at 120°F and 95-100% RH.

2.2.1 Hysol EA 9309.3NA

Results for tensile lap shear and floating roller peel testing of EA 9309.3NA are shown in Table 5. For specimens with less than 100% cohesive failure modes, interfacial failure occurred between the primer and adhesive in PAA/BR 127 specimens and between the adhesive and aluminum on the other specimens. When EA 9309.3NA was cured at elevated temperature, the scuff-sand/solvent wipe process exhibited reduced bond strengths and low percentages of cohesive failure. Most noticeably, it can be seen that EA 9309.3NA exhibited high peel strength when bonded to adherends prepared using PAA/BR 127, however, the peel strength is drastically reduced when bonded to the scuff-sand/solvent wipe surface. At all temperatures, the nylon-pad/sol-gel process provided lap shear strengths comparable with those obtained with PAA/BR 127, but exhibited lower percentages of cohesive failure, especially when tested at 160°F and 180°F. However, the peel strengths of the specimens prepared with the nylon-pad/sol-gel process were reduced when compared to PAA/BR 127, and they exhibited a lower percentage of cohesive failure.

Overall, when ambient-temperature cure is compared to elevated-temperature cure, the lap shear strength of the adhesive is drastically reduced as test temperature increases. The nylon-pad/sol-gel process yields lap shear and peel strengths similar to those of PAA/BR 127 tests, but specimens exhibit very low percentages of cohesive failure when tested in lap shear at elevated temperature.

Table 5: Effect of Surface Preparation on Mechanical Properties of EA 9309.3NA

Surface Preparation		Lap Shear Strength (psi) (% cohesive failure)			Peel Strength (pli)
		70°F	160°F	180°F	70°F
ET	PAA / BR 127	4318* (95% co)	2295* (99% co)	1029* (89% co)	54.4* (88% co)
ET	Scuff-sand / solvent wipe	4272* (32% co)	1971* (33% co)	751* (0% co)	8.8* (0% co)
ET	Nylon-pad / sol-gel	4705* (91% co)	2528* (76% co)	1028* (5% co)	43.8* (58% co)
AT	PAA / BR 127 (pp)**	3819 (95% co)	615 (98% co)	439 (99% co)	50.9 (100% co)
AT	Nylon-pad / sol-gel	3500 (87% co)	580 (3% co)	521 (23% co)	50.9 (100% co)

AT: Ambient Temperature cure ** Average of 10 specimens*
ET: Elevated Temperature cure *** (pp): cured under positive pressure*

Results of the EA 9309.3NA wedge tests are shown in Table 6. The PAA/BR 127 specimens exhibited cohesive failure modes and the shortest cracks. Any interfacial failure detected on

PAA/BR 127 specimens occurred between the adhesive and primer. The nylon-pad/sol-gel specimens exhibited higher percentages of cohesive failure and shorter crack lengths than the scuff-sand/solvent wipe specimens. The interfacial failure modes present in the scuff-sand solvent wipe and nylon-pad/sol-gel specimens occurred between the adhesive and aluminum.

For a given EA 9309.3NA cure cycle, the nylon-pad/sol-gel surface preparation resulted in bonds with similar strengths to those using PAA/BR 127, but with smaller amounts of cohesive failure. The nylon-pad/sol-gel process did not provide the same level of durability as PAA/BR 127 as evidenced by the wedge tests. The nylon-pad/sol-gel surface preparation outperformed scuff-sand/solvent wipe in all tests conducted with EA 9309.3NA.

Table 6: Effect of Surface Preparation on Wedge Test Results Using EA 9309.3NA

Surface Preparation		Initial (in)	Cummulative Crack Growth (in)							Total (in)	Failure Mode*
			1 hr	8 hrs	24 hrs	7 days	14 days	21 days	28 days		
ET	PAA / BR127	1.45	0.06	0.14	0.19	0.43	0.51	0.58	0.63	2.08	85% co
ET	PAA / BR127	1.39	0.04	0.13	0.20	0.43	0.54	0.61	0.66	2.05	97% co
ET	Scuff sand / acetone wipe	1.40	0.68	0.72	0.76	0.93	1.08	1.14	1.15	2.55	0% co
ET	Scuff sand / acetone wipe	1.50	1.16	1.17	1.17	1.19	1.21	1.23	1.25	2.75	0% co
ET	Nylon pad / sol-gel	1.40	0.07	0.21	0.32	0.65	0.78	0.89	0.99	2.39	28% co
ET	Nylon pad / sol-gel	1.46	0.10	0.18	0.21	0.36	0.46	0.53	0.58	2.04	42% co
AT	PAA / BR127 (pp)	1.49	0.12	0.33	0.44	0.62	0.74	0.83	0.88	2.37	100% co
AT	Scuff sand / acetone wipe	1.42	0.45	0.47	0.51	0.56	0.64	0.76	0.83	2.25	0% co
AT	Nylon pad / sol-gel	1.55	0.13	0.30	0.38	0.86	1.06	1.08	1.10	2.65	74% co

*co: cohesive failure

2.2.2 Hysol EA 9320NA

Results for tensile lap shear and floating roller peel testing of EA 9320NA are shown in Table 7. The small amount of interfacial failure exhibited by some of the PAA/BR 127 specimens occurred between the primer and adhesive. For the other specimens, interfacial failure occurred between the aluminum and adhesive. The scuff-sand/solvent wipe process exhibited significantly reduced lap shear strengths and failure modes. Peel strength for specimens prepared with the EA 9320NA was almost undetectable when using the scuff-sand/solvent wipe process. Lap shear specimens prepared with the nylon-pad/sol-gel process exhibited very similar bond strengths and failure modes to those of PAA/BR 127-prepared specimens when cured at ambient temperature or elevated temperature and tested at ambient temperature. There was a

reduction in the percent cohesive failure for nylon-pad/sol-gel lap shear specimens cured and tested at elevated temperature.

Table 7: Effect of Surface Preparation on Mechanical Properties of EA 9320NA

Surface Preparation		Lap Shear Strength (psi) (% cohesive failure)			Peel Strength (pli)
		70°F	160°F	180°F	70°F
ET	PAA / BR 127	5197 (98% co)	3147 (100% co)	1812 (97% co)	27.3 (100% co)
ET	Scuff-sand / solvent wipe	3366 (60% co)	1954 (23% co)	1040 (-0-% co)	2.7 (-0-% co)
ET	Nylon-pad / sol-gel	4807 (99% co)	2970 (80% co)	1667 (50% co)	25.1 (100% co)
AT	PAA / BR 127 (pp)	4620 (99% co)	1126 (100% co)	843 (100% co)	23.9 (100% co)
AT	Nylon-pad / sol-gel	3992 (100% co)	1018 (100% co)	734 (100% co)	23.2 (100% co)

Results of the EA 9320NA wedge tests are shown in Table 8. The PAA/BR 127 and nylon-pad/sol-gel specimens exhibited excellent failure modes and the shortest cracks. The scuff-sand/solvent wipe specimens resulted in gross adhesive failure at the aluminum-adhesive interface within 24 hours of testing. Overall, the nylon-pad/sol-gel process yielded excellent results in mechanical and durability testing when used with EA 9320NA adhesive, even though the process did not include a primer.

Table 8: Effect of Surface Preparation on Wedge Test Results Using EA 9320NA

Surface Preparation		Initial (in)	Cummulative Crack Growth (in)							Total (in)	Failure Mode
			1 hr	8 hrs	24 hrs	7 days	14 days	21 days	28 days		
ET	PAA / BR127	1.63	0.05	0.06	0.06	0.06	0.11	0.11	0.11	1.74	100% co
ET	Scuff sand / acetone wipe	1.63	0.49	0.60	0.63	0.69	0.73	0.74	0.75	2.38	-0-% co
ET	Nylon pad / sol-gel	1.64	0.04	0.10	0.10	0.16	0.22	0.26	0.26	1.90	95% co
AT	PAA / BR127 (pp)	1.77	0.16	0.16	0.16	0.18	0.19	0.19	0.20	1.98	100% co
AT	Nylon pad / sol-gel	1.73	0.10	0.10	0.10	0.11	0.12	0.13	0.14	1.88	100% co

2.2.3 Hysol EA 9330.3

Results for tensile lap shear and floating roller peel testing of EA 9330.3 are shown in Table 9. Although the PAA/BR 127 specimens' failure modes were mostly cohesive, some exhibited partial failures at the primer-adhesive interface. Interfacial failure exhibited by other specimens was between the aluminum and primer. The scuff-sand/solvent wipe process with EA 9330.3 did not result in the significant reductions in ambient-temperature lap shear and peel strengths that were observed using this process with the other adhesives. However, scuff-sand solvent wipe

specimens experienced a large reduction in lap shear strength when tested at elevated temperature. These specimens also resulted in complete interfacial failure between the adhesive and metal.

With EA 9330.3 cured at ambient temperature, the nylon-pad/sol-gel specimens yielded similar strengths and failure modes as PAA/BR 127 specimens. However, when the cure of the adhesive was accelerated with heat, specimens prepared with the nylon-pad/sol-gel process exhibited reduced amounts of cohesive failure when tested at elevated temperature, although their strengths are very consistent with that of the PAA/BR 127 specimens. The specimens prepared with the nylon-pad/sol-gel process again outperform those prepared with the scuff-sand/solvent wipe process.

Table 9: Effect of Surface Preparation on Mechanical Properties of EA 9330.3

Surface Preparation		Lap Shear Strength (psi) (% cohesive failure)			Peel Strength (pli)
		70°F	160°F	180°F	70°F
ET	PAA / BR 127	5000 (93% co)	729 (88% co)	662 (81% co)	48.1 (100% co)
ET	Scuff-sand / solvent wipe	4533 (82% co)	441 (-0-% co)	378 (-0-% co)	39.3 (70% co)
ET	Nylon-pad / sol-gel	4138 (99% co)	903 (68% co)	666 (48% co)	42.7 (100% co)
AT	PAA / BR 127 (pp)	4414 (100% co)	764 (100% co)	582 (98% co)	38.4 (100% co)
AT	Nylon-pad / sol-gel	4262 (100% co)	628 (89% co)	482 (95% co)	36.7 (100% co)

Results of the EA 9330.3 wedge tests are shown in Table 10. PAA/BR 127 test specimens exhibited excellent failure modes and the shortest cracks. The scuff-sand/solvent wipe specimens resulted in interfacial failure after 28 days of testing. The nylon-pad/sol-gel specimens exhibited a mixed failure mode and longer cracks than the PAA /BR 127 specimens. Overall, the nylon-pad/sol-gel process yielded better results than the scuff-sand/solvent wipe process, but lacked the wedge test durability that PAA/BR 127 provided with EA 9330.3.

Table 10: Effect of Surface Preparation on Wedge Test Results Using EA 9330.3

Surface Preparation		Initial (in)	Cummulative Crack Growth (in)							Total (in)	Failure Mode
			1 hr	8 hrs	24 hrs	7 days	14 days	21 days	28 days		
ET	PAA / BR127	1.49	0.22	0.53	0.79	1.21	1.27	1.33	1.37	2.86	87% co
ET	Scuff sand / acetone wipe	1.34	0.29	1.04	1.64	2.45	2.51	2.51	2.56	3.90	-0-% co
ET	Nylon pad / sol-gel	1.41	0.16	0.92	1.32	1.66	1.71	1.75	1.75	3.16	40% co
AT	PAA / BR127 (pp)	1.58	0.55	0.95	1.06	1.25	1.32	1.36	1.41	2.98	100% co
AT	Nylon pad / sol-gel	1.61	0.47	0.93	1.35	1.92	1.98	1.98	1.99	3.60	31% co

2.2.4 Hysol EA 9394

Results for tensile lap shear and floating roller peel testing of EA 9394 are shown in Table 11. Where failure modes are not cohesive, failure occurred between the primer and adhesive in PAA/BR 127 specimens and between the adhesive and aluminum in the other specimens. EA 9394 specimens exhibited excellent properties when tested at elevated temperature. The EA 9394 also yielded excellent properties when cured at ambient temperature as compared to the other paste adhesives which tended to lose elevated-temperature strength rapidly when cured at ambient. The peel strengths obtained for EA 9394 were slightly high when compared to the manufacturer's published value of 20 pli. These peel strengths are also considerably higher than published results of 10 pli found in previous work conducted at UDRI[12]. As shown with other paste adhesives, the scuff-sand/solvent wipe surface preparation yielded interfacial failures and lower strengths compared to specimens prepared with PAA/BR 127 and the nylon-pad/sol-gel processes. The poor performance of the scuff-sand/solvent wipe surface preparation was especially evident in the floating roller peel results.

Table 11: Effect of Surface Preparation on Mechanical Properties of EA 9394

Surface Preparation		Lap Shear Strength (psi) (% cohesive failure)			Peel Strength (pli)
		70°F	160°F	180°F	70°F
ET	PAA / BR 127	3818 (93% co)	2926 (95% co)	2798 (98% co)	23.2 (89% co)
ET	Scuff-sand / solvent wipe	2823 (31% co)	2210 (93% co)	2282 (74% co)	3.3 (-0-% co)
ET	Nylon-pad / sol-gel	3829 (46% co)	2379 (88% co)	2301 (80% co)	16.7 (79% co)
AT	PAA / BR 127 (pp)	4076 (94% co)	2522 (100% co)	2697 (100% co)	25.6 (97% co)
AT	Nylon-pad / sol-gel	3234 (97% co)	2043 (93% co)	2029 (85% co)	25.2 (95% co)

Results of the EA 9394 wedge tests are shown in Table 12. Initial cracks in the scuff-sand/solvent wipe specimens were at the aluminum-adhesive interface. The initial crack lengths for the scuff-sand/solvent wiped specimens were approximately 2.76 inches as compared to cohesive (within the adhesive) initial cracks exhibited with the PAA/BR 127 and nylon-pad/sol-gel specimens of approximately 1.80 inches. After 28 days in humidity, specimens prepared with the nylon-pad/sol-gel process had smaller crack lengths than the specimens prepared using the scuff-sand/solvent wipe process. The nylon-pad/sol-gel process was not able to achieve the same performance as PAA/BR 127, as shown by the larger crack lengths and lesser amounts of cohesive failure.

Table 12: Effect of Surface Preparation on Wedge Test Results Using EA 9394

Surface Preparation		Initial (in)	Cummulative Crack Growth (in)							Total (in)	Failure Mode
			1 hr	8 hrs	24 hrs	7 days	14 days	21 days	28 days		
ET	PAA / BR127	1.89	0.13	0.14	0.16	0.20	0.24	0.24	0.25	2.14	100% co
ET	Scuff sand / acetone wipe	2.76	0.05	0.11	0.11	0.18	0.20	0.21	0.21	2.97	-0-% co
ET	Nylon pad / sol-gel	1.75	0.12	0.23	0.48	0.62	0.69	0.69	0.75	2.50	44& co
AT	PAA / BR127 (pp)	1.88	0.14	0.14	0.15	0.21	0.23	0.23	0.27	2.15	100% co
AT	Nylon pad / sol-gel	1.81	0.22	0.23	0.27	0.48	0.57	0.62	0.70	2.52	38% co

2.2.5 Hysol EA 9396

Results for tensile lap shear and floating roller peel testing of EA 9396 are shown in Table 13. Again, any interfacial failure exhibited occurred between the primer and adhesive in PAA/BR 127 specimens and between the adhesive and aluminum with the other specimens. In general, higher strengths and better failure modes were obtained when curing EA 9396 at ambient temperature versus the accelerated elevated-temperature cure. This is particularly evident in the peel results. Scuff-sand/solvent wipe specimens resulted in complete adhesive failure between the aluminum and adhesive in all cases. In fact, the peel specimens were not tested because they fell apart during machining. The nylon-pad/sol-gel surface preparation yielded much better results compared to the scuff-sand/solvent wipe process, but did not match the strengths or failure modes achieved by specimens prepared with PAA/BR 127.

Table 13: Effect of Surface Preparation on Mechanical Properties of EA 9396

Surface Preparation		Lap Shear Strength (psi) (% cohesive failure)			Peel Strength (pli)
		70°F	160°F	180°F	70°F
ET	PAA / BR 127	3766 (62% co)	3021 (89% co)	2166 (38% co)	13.9 (95% co)
ET	Scuff-sand / solvent wipe	1287 (-0-% co)	689 (-0-% co)	613 (-0-% co)	Broke during machining
ET	Nylon-pad / sol-gel	3550 (28% co)	1736 (30% co)	2148 (24% co)	9.6 (50% co)
AT	PAA / BR 127 (pp)	5248 (98% co)	3288 (100% co)	2993 (91% co)	24.5 (100% co)
AT	Nylon-pad / sol-gel	3380 (99% co)	1795 (54% co)	1778 (26% co)	22.0 (85% co)

Results of the EA 9396 wedge tests are shown in Table 14. The nylon-pad/sol-gel surface preparation yielded larger crack growths than did PAA/BR 127. Specimens fabricated with the nylon-pad/sol-gel process resulted in complete adhesive failure between the aluminum and adhesive when EA 9396 was cured at ambient temperature. Initial cracks in the scuff-sand/solvent wipe specimens were at the aluminum-adhesive interface. The initial cracks were

approximately 4.50 inches in length compared to cohesive initial cracks of 1.8 inches. The scuff-sand/solvent wipe specimens failed prior to the one-hour reading. Overall, the scuff-sand/solvent wipe specimens exhibited lower bond strengths, less durability, and complete adhesive failures at all testing conditions. The nylon-pad/sol-gel process provided better strength and durability than the scuff-sand/solvent wipe process, but was unable to meet the performance provided by PAA/BR 127.

Table 14: Effect of Surface Preparation on Wedge Test Results Using EA 9396

Surface Preparation		Initial (in)	Cummulative Crack Growth (in)							Total (in)	Failure Mode
			1 hr	8 hrs	24 hrs	7 days	14 days	21 days	28 days		
ET	PAA / BR127	1.89	0.11	0.17	0.17	0.22	0.33	0.35	0.40	2.29	83% co
ET	Scuff sand / acetone wipe	4.46	Fell apart by 1 hour reading							>4.46	-0-% co
ET	Nylon pad / sol-gel	1.97	0.16	0.54	0.66	0.80	0.92	0.92	0.92	2.89	88% co
AT	PAA / BR127 (pp)	1.74	0.13	0.20	0.23	0.34	0.39	0.44	0.45	2.19	100% co
AT	Nylon pad / sol-gel	1.90	0.14	0.73	1.19	1.55	1.58	1.60	1.64	3.54	3% co

2.2.6 3M Company EC 2615

Results for tensile lap shear and floating roller peel testing of EC 2615 are shown in Table 15. Again, any interfacial failure occurred between the primer and adhesive in PAA/BR 127 specimens and between the adhesive and aluminum on the other specimens. EC 2615 yielded better strength at elevated temperature when cured at elevated temperature versus cured at ambient temperature, but exhibited large amounts of interfacial failure, even in PAA/BR 127 specimens. In fact, PAA/BR 127 peel specimens exhibited complete interfacial failure between the primer and adhesive when cured at elevated temperature. However, when cured at ambient temperature, PAA/BR 127 specimens yielded higher percentages of cohesive failure, and much higher peel strengths. The detriment to ambient cure was the reduction in elevated-temperature lap shear strength. Scuff-sand/solvent wipe specimens resulted in complete adhesive failure between the aluminum and adhesive in all cases. The nylon-pad/sol-gel surface preparation produced much better results than the scuff-sand/solvent wipe process, but did not match the strengths or failure modes achieved with specimens prepared with PAA/BR 127.

Table 15: Effect of Surface Preparation on Mechanical Properties of EC 2615

Surface Preparation		Lap Shear Strength (psi) (% cohesive failure)			Peel Strength (pli)
		70°F	160°F	180°F	70°F
ET	PAA / BR 127	4255 (62% co)	2195 (56% co)	2179 (30% co)	46.9 (-0-% co)
		5924 (8% co)	2390 (62% co)	1372 (36% co)	58.0 (10% co)
	Scuff-sand / solvent wipe	3710 (-0-% co)	2283 (-0-% co)	1148 (-0-% co)	1.8 (-0-% co)
	Nylon-pad / sol-gel	4819 (6% co)	2195 (14% co)	1356 (2% co)	38.0 (-0-% co)
AT	PAA / BR 127 (pp)	4870 (11% co)	801 (90% co)	714 (39% co)	77.0 (90% co)
	Nylon-pad / sol-gel	4244 (46% co)	1053 (-0-% co)	444 (-0-% co)	60.4 (70% co)

Results of the EC 2615 wedge tests are shown in Table 16. PAA/BR 127 specimens exhibited interfacial failure between the primer and adhesive. Interfacial failure occurred at the metal-adhesive interface on all other specimens. Overall, more interfacial failure occurred in specimens cured at elevated temperature than those cured at ambient temperature. Scuff-sand/solvent wipe specimens resulted with initial cracks occurring completely at the aluminum-adhesive interface and were removed from humidity after 7 days due to excessive crack length. The initial cracks were approximately 2.53 inches in length compared to cohesive initial cracks of 1.5 inches. Overall, the scuff-sand/solvent wipe specimens exhibited lower bond strengths, less durability, and complete adhesive failures at all testing conditions. The nylon-pad/sol-gel process provided better strength and durability than the scuff-sand/solvent wipe process, but was unable to meet the performance provided by PAA/BR 127.

Table 16: Effect of Surface Preparation on Wedge Test Results Using EC 2615

Surface Preparation		Initial (in)	Cummulative Crack Growth (in)							Total (in)	Failure Mode
			1 hr	8 hrs	24 hrs	7 days	14 days	21 days	28 days		
ET	PAA / BR127	1.52	0.00	0.03	0.04	0.16	0.25	0.31	0.38	1.90	64% co
	Scuff sand / acetone wipe	2.53	1.46	1.47	1.47	1.47	removed due gross failure			4.00	-0-% co
	Nylon pad / sol-gel	1.60	0.01	0.06	0.15	0.55	0.76	0.93	1.07	2.67	5% co
AT	PAA / BR127 (pp)	0.97	0.09	0.31	0.38	0.56	0.61	0.66	0.70	1.66	99% co
	Nylon pad / sol-gel	0.93	0.14	0.28	0.36	0.64	0.74	0.82	0.86	1.79	43% co

2.2.7 3M Company EC 3333

Results for tensile lap shear and floating roller peel testing of EC 3333 are shown in Table 17. All interfacial failure occurred between the primer and adhesive in PAA/BR 127 specimens and between the adhesive and aluminum on the other specimens. As was the case for EC 2615, EC 3333 yielded better lap shear strength at elevated temperature when cured at elevated

temperature, but exhibited large amounts of interfacial failure, even in PAA/BR 127 specimens. PAA/BR 127 peel specimens exhibited complete interfacial failure between the primer and adhesive when cured at elevated temperature. Curing at ambient temperature did not seem to improve the amount of cohesive failure detected in lap shear specimens but drastically improved the strength and failure modes in peel tests. PAA/BR 127 specimens yielded the best lap shear and peel strengths when cured at elevated temperature. Scuff-sand/solvent wipe specimens resulted in reduced bond strengths, especially in peel, and complete adhesive failure between the aluminum and adhesive in all cases. The nylon-pad/sol-gel surface preparation produced much better results than the scuff-sand/solvent wipe process, but did not match the strengths or failure modes achieved with specimens prepared with PAA/BR 127.

Table 17: Effect of Surface Preparation on Mechanical Properties of EC 3333

Surface Preparation		Lap Shear Strength (psi) (% cohesive failure)			Peel Strength (pli)
		70°F	160°F	180°F	70°F
ET	PAA / BR 127	4960 (20% co)	3500 (52% co)	2180 (6% co)	44.3 (-0-% co)
ET	PAA / BR 127	5489 (6% co)	3059 (20% co)	1439 (20% co)	55.4 (-0-% co)
ET	Scuff-sand / solvent wipe	4134 (-0-% co)	1825 (-0-% co)	600 (-0-% co)	2.6 (-0-% co)
ET	Nylon-pad / sol-gel	4894 (12% co)	2525 (23% co)	964 (-0-% co)	38.3 (-0-% co)
AT	PAA / BR 127 (pp)	3477 (-0-% co)	1087 (-0-% co)	453 (27% co)	75.9 (100% co)
AT	Nylon-pad / sol-gel	5281 (57% co)	877 (-0-% co)	729 (-0-% co)	57.8 (80% co)

Results of the EC 2615 wedge tests are shown in Table 18. PAA/BR 127 specimens exhibited interfacial failure between the primer and adhesive. Interfacial failure occurred at the metal-adhesive interface on all other specimens.

All specimens failed adhesively except for the PAA/BR 127 specimens. Specimens prepared with the scuff-sand/solvent wipe procedure exhibited initial cracks at the aluminum-adhesive interface. These cracks were approximately 2.55 inches in length compared to cohesive initial cracks of 1.4 inches. Overall, the scuff-sand/solvent wipe specimens produced lower bond strengths, less durability, and complete adhesive failures at all testing conditions. The nylon-pad/sol-gel process provided better strength and durability than the scuff-sand/solvent wipe process, but was unable to meet the performance provided by PAA/BR 127.

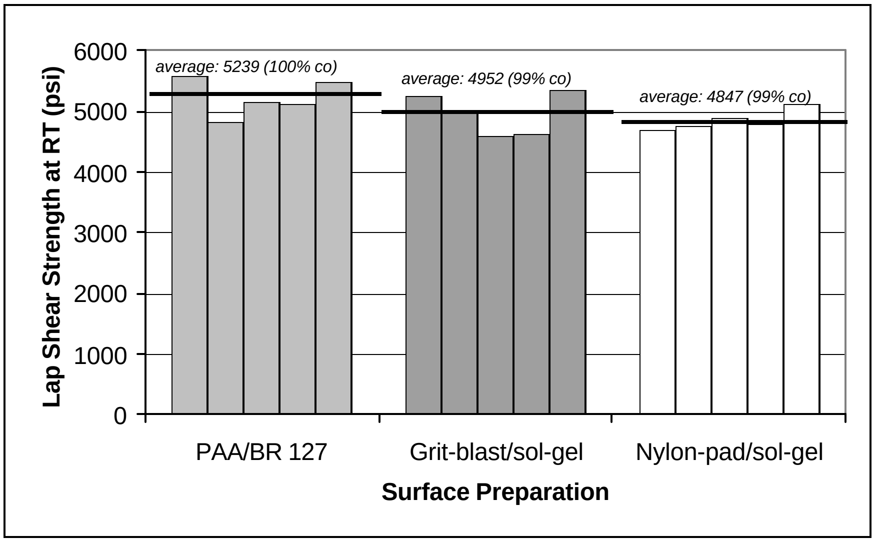
Table 18: Effect of Surface Preparation on Wedge Test Results Using EC 3333

Surface Preparation		Initial (in)	Cummulative Crack Growth (in)							Total (in)	Failure Mode
			1 hr	8 hrs	24 hrs	7 days	14 days	21 days	28 days		
ET	PAA / BR127	1.40	0.01	0.04	0.08	0.25	0.38	0.46	0.51	1.91	47% co
	Scuff sand / acetone wipe	2.55	0.68	0.68	0.68	0.72	0.74	0.84	0.86	3.41	-0-% co
	Nylon pad / sol-gel	1.21	0.06	0.16	0.24	0.45	0.57	0.67	0.74	1.95	-0-% co
AT	PAA / BR127 (pp)	1.07	0.08	0.36	0.47	0.64	0.66	0.72	0.76	1.83	76% co
	Nylon pad / sol-gel	1.01	0.16	0.33	0.41	0.64	0.78	0.84	0.91	1.92	-0-% co

2.3 Repeatability Assessment

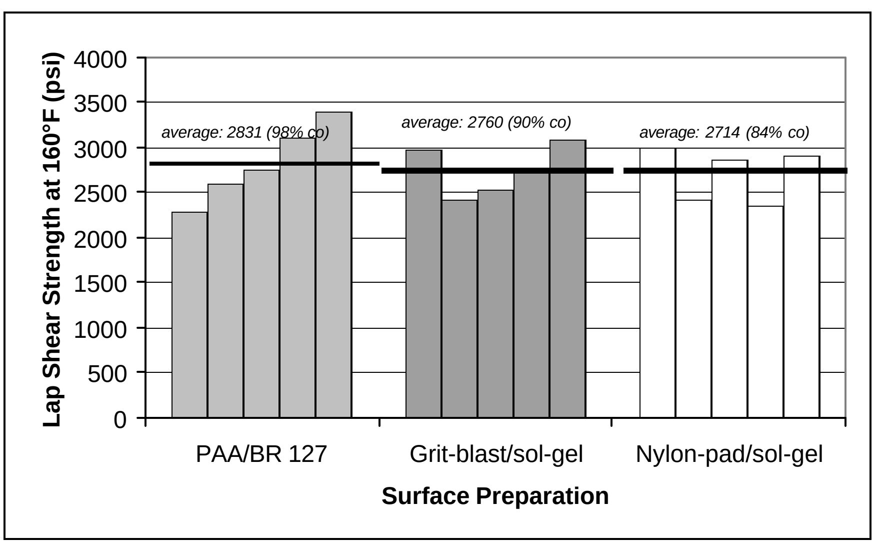
In order to determine the repeatability of the nylon-pad/sol-gel surface preparation using EA 9320NA, an assessment was conducted using tensile lap shear and wedge testing. Five wedge test panels (tested at 120°F & 95-100% RH) and ten lap shear panels (five tested at 70°F and five tested at 160°F) were fabricated with each of the following surface preparations: grit-blast/sol-gel[13], nylon-pad/sol-gel, and PAA/BR 127 (control). Neither sol-gel surface preparation included an adhesive bond primer. Al 7075-T6 adherends were used in this assessment as opposed to Al 2024-T3. Adherends were degreased by wiping with lint-free cloths moistened with acetone. Nylon-pad abraded panels were deoxidized with 3-inch diameter 3M Company Scotch-Brite™ Roloc™ medium pads using a 20,000-RPM nitrogen-driven rotary grinder. Grit-blasted panels were blasted with 50-micron Al_2O_3 grit. All panels were blown with 35 psi nitrogen to remove residual grit or residue prior to application of sol-gel solution. Sol-gel solution was brush applied so the surface was kept wet for 3 minutes. Panels were allowed to dry at ambient laboratory conditions (70°F and 60% RH) for 30 minutes prior to application of adhesive. Adhesive was applied on both bonding surfaces and a random-mat polyester scrim cloth was used for bondline control. Panels were cured for 2 hours at 150°F and 15 inches of Hg vacuum pressure. Results of the ambient-temperature tensile lap shear testing are shown in Figure 1. It can be seen from Figure 1 there is little change in lap shear strength or failure mode associated with surface preparation, and the results are very repeatable as shown with five separate panels (25 specimens).

Figure 1: Effect of Surface Preparation on Ambient-Temperature Lap Shear Strength Using EA 9320NA



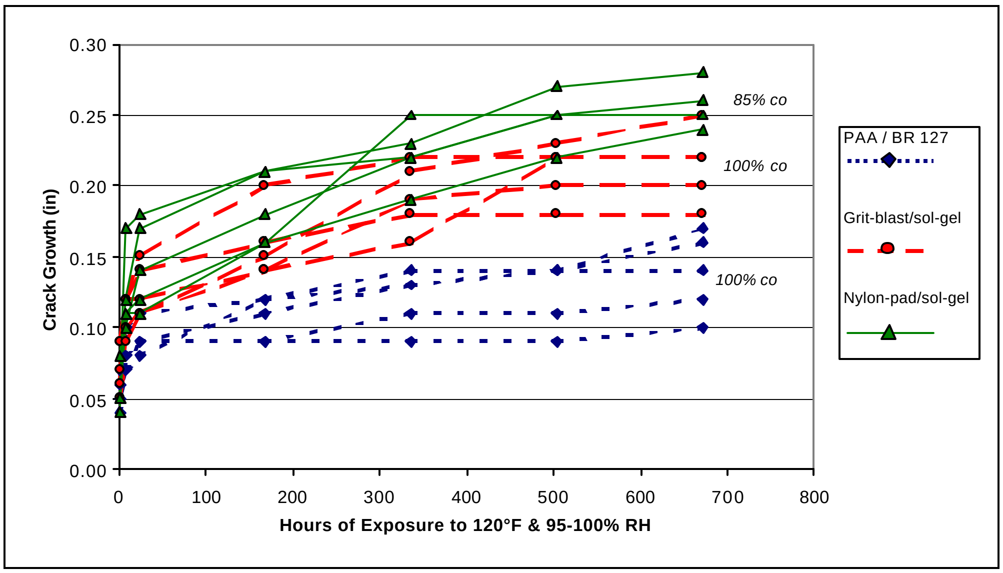
Results of the lap shear testing conducted at 160°F are shown in Figure 2. The lap shear strength of the bond drops with increasing temperature. However, the average strengths of the bonds prepared with the different surface preparations are relatively the same. There is a drop of percent cohesive failure mode when using both the grit-blast and nylon-pad sol-gel processes when compared to PAA/BR 127. The small amount of adhesive failure occurred between the aluminum and adhesive. Results appear to be reproducible within the five panels.

Figure 2: Effect of Surface Preparation on Lap Shear Strength at 160°F Using EA 9320NA



Results of the wedge testing are shown in Figure 3. The nylon-pad/sol-gel process did not result in the same failure modes as the grit-blast/sol-gel and PAA/BR 127 processes. However, there did appear to be repeatability within each of the processes. Since the data using the nylon pad/sol-gel process without primer led to less than the desired (100% cohesive failure), the tests were repeated varying the coarseness of the 3M Roloc™ pads. Two panels were fabricated per testing condition with both coarse and medium grade Roloc™ pads. Results of the ambient-temperature lap shear testing are shown in Figure 4. Results of the lap shear testing conducted at 160°F are shown in Figure 5.

Figure 3: Effect of Surface Preparation on Wedge Test Results Using EA 9320NA



The ambient-temperature lap shear data from Figure 4 do not seem to distinguish a difference between the different abrasive pads. Failure modes were all cohesive. However, changes in failure modes were detected when tested at 160°F (Figure 5). Use of medium Roloc™ pads resulted in 84% cohesive failure in the first run (data from Figure 2), and these data was verified in the second run with an average of 86% cohesive failure. When using coarse pads, the failure mode increased to 99% cohesive. There was little difference in strength noticed between specimens prepared with different grades of nylon pads even though there were changes in the amount of cohesive failure present.

Figure 4: Effect of Nylon-Pad Coarseness on Ambient-Temperature Lap Shear Strength Using EA 9320NA

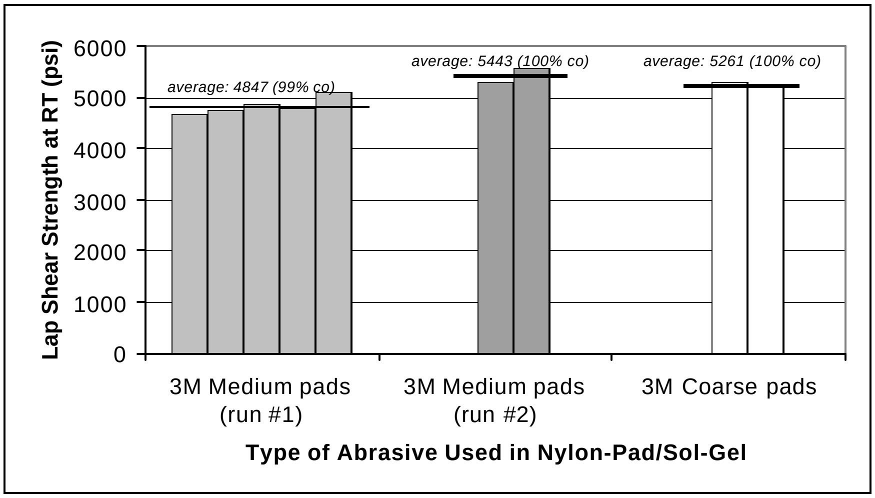
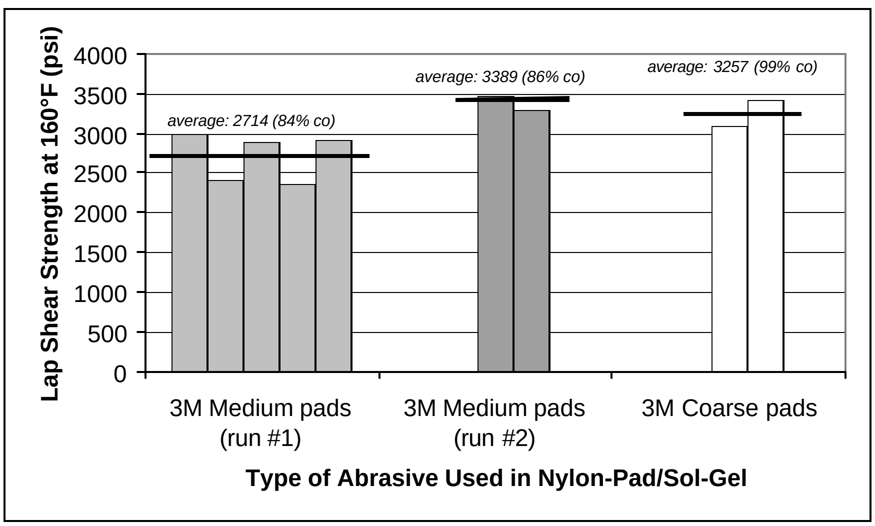
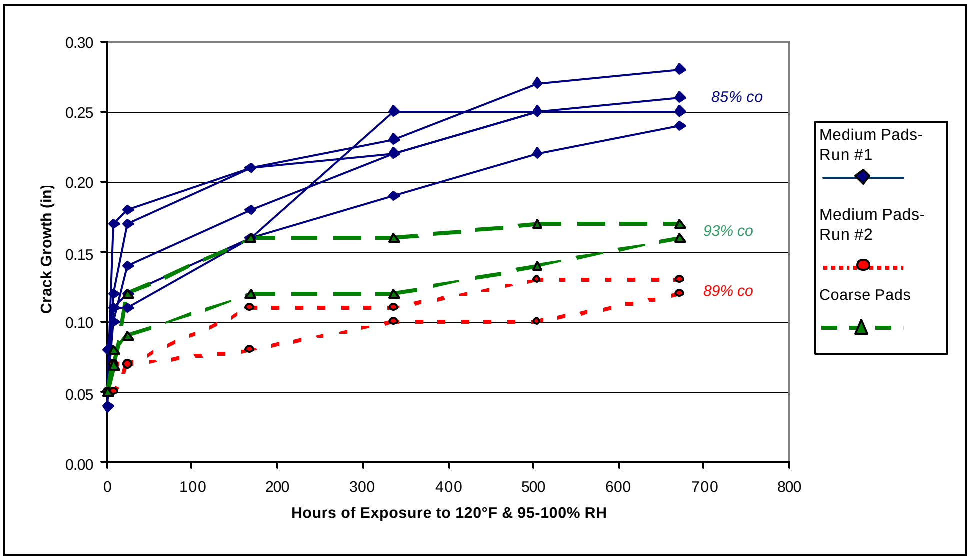


Figure 5: Effect of Nylon-Pad Coarseness on Lap Shear Strength at 160°F Using EA 9320NA



Results of the wedge testing are shown in Figure 6. The failure modes seem consistent when using medium pads when comparing Run #1 to Run #2. In both cases, failure modes were about 85-89% cohesive. However, the crack growth was smaller in Run #2 when using medium pads compared to Run #1. When using coarse pads, the amount of cohesive failure increased a small amount to an average value of 93% cohesive.

Figure 6: Effect of Nylon-Pad Coarseness on Wedge Test Results Using EA 9320NA



3 DISCUSSION

3.1 Determination of Baseline Adhesive Properties

As was expected, the paste adhesives evaluated exhibited a range of properties. Lap shear and peel tests, conducted on Al 2024-T3 using PAA/BR 127, yielded high percentages of cohesive failure with the Hysol adhesives and low percentages of cohesive failure with the 3M adhesives, in most cases. Lap shear testing at 180°F was the one test condition that tended to result in lower percentages of cohesive failure with the Hysol adhesives. This was somewhat unexpected since the lap shear strength of an adhesive tends to decrease with increasing temperature. This should increase the likelihood of failure within the adhesive layer. However, experimental results show that the elevated temperature drove the failure to the interface with several adhesives tested at 180°F. EA 9394 and EA 9396 provided the best strengths when tested at elevated temperature. This was especially true when curing the paste adhesives at ambient temperature. All the paste adhesives experienced a sharp reduction in lap shear strength at elevated temperature due to the ambient cure, except EA 9394 and EA 9396. This was as expected since these adhesives were formulated for high-temperature service with low-temperature cure. The trade-off is their reduced peel strengths.

PAA/BR 127 wedge test results were generally as expected, with high percentages of cohesive failure. EA 9330.3 specimens exhibited roughly 70% cohesive failure with some interfacial failure occurring at the adhesive-primer interface. The other Hysol adhesives exhibited high percentages of cohesive failure. However, the 3M adhesives exhibited extremely small percentages of cohesive failure with failure occurring at the adhesive-primer interface.

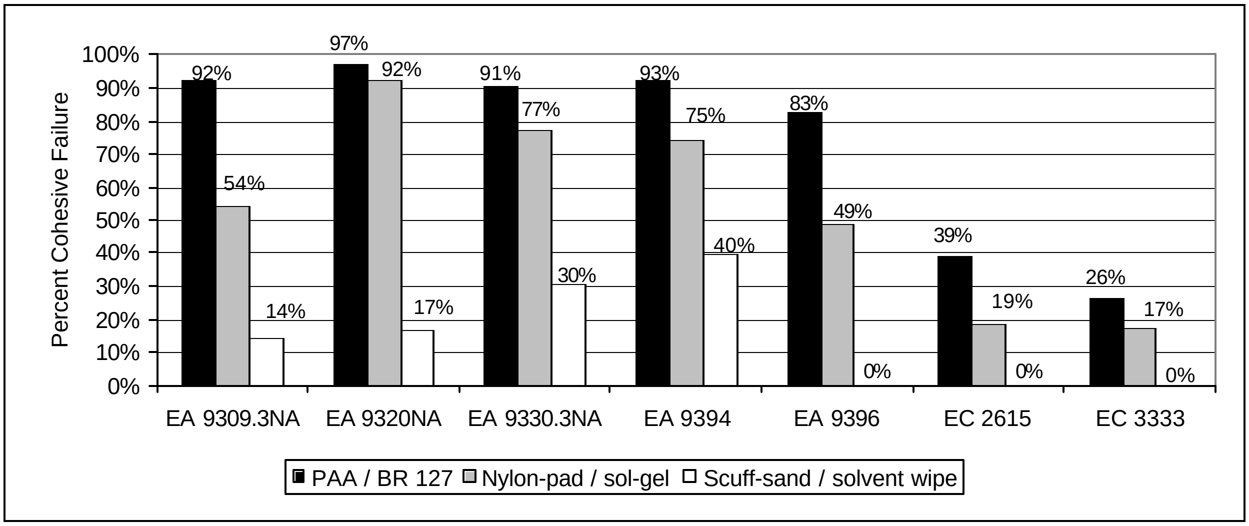
The 3M adhesives (EC 2615 and EC 3333) produced extremely high percentages of adhesive failure mode at the primer-adhesive interface in lap shear, peel and wedge tests conducted using Al 2024-T3 treated with PAA/BR 127. This was unexpected since PAA/BR 127 is the premier surface preparation for aluminum and typically provides cohesive failure modes for lap shear, peel, and wedge testing. It is unknown why the 3M adhesives exhibited such low percentages of cohesive failure when compared to the Hysol adhesives, but it is a concern for bond durability. One possible cause of failure is some type of incompatibility between the 3M adhesives and the Cytec BR 127 primer. This was not further investigated in this study.

3.2 Evaluation of Nylon-Pad/Sol-Gel Surface Preparation

In order to estimate the overall effect of surface preparation for each adhesive, a chart comparing the average percent cohesive failure for all lap shear, peel, and wedge test specimens for each process investigated in the surface preparation evaluation is shown in Figure 7. This was a qualitative comparison based solely on failure modes. Since it includes lap shear, peel, and wedge data, it is possible that one test, such as the wedge test, can have a large influence on the results. The PAA/BR 127 process yields the highest average amount of cohesive failure for each adhesive. This process was also the only one that included an adhesive bond primer. The nylon-pad/sol-gel process yields a higher average amount of cohesive failure than the scuff-sand/solvent wipe process for every adhesive. For all adhesives tested, the nylon-pad/sol-gel process provided a reduced amount of cohesive failure when compared to PAA/BR 127. The reason for the better performance obtained with EA 9320NA was not determined. However, when using EA 9320NA, the nylon-pad/sol-gel process yielded an average of roughly 92% cohesive failure mode, when compared to the 97% cohesive mode routinely achieved with PAA/BR 127. This was the best failure mode achieved using the nylon-pad/sol-gel process with any of the paste adhesives. Specimens prepared with the nylon-pad/sol-gel surface preparation

performed very similarly to those prepared with PAA/BR 127 when bonded with EA 9320NA, as detailed in Section 2.2.2. The 3M adhesives exhibited extremely low percentages of cohesive failure for all surface preparations when compared to the Hysol adhesives.

Figure 7: Average Percent Cohesive Failure versus Surface Preparation for Various Adhesives



3.3 Evaluation of the Repeatability Assessment

Ambient-temperature lap shear results using EA 9320NA adhesive did not distinguish differences between PAA, grit-blast/sol-gel or nylon-pad/sol-gel surface preparations. All specimens failed cohesively with very repeatable strengths. All five panels (25 specimens) per surface preparation were comparative with each other as shown in Figure 1. Lap shear strength at 160°F did reveal differences in failure mode between surface preparation (Figure 2), even though the strengths were consistent. PAA specimens exhibited cohesive failure while grit-blast/sol-gel specimens exhibited ~90% cohesive failure and nylon-pad/sol-gel specimens exhibited ~84% cohesive failure. Identically prepared panels performed similarly and led to very reproducible results with all three evaluated surface preparations when tested for lap shear strength at 160°F. Wedge tests (Figure 3) show that PAA and grit-blast/sol-gel specimens exhibit nearly 100% cohesive failure modes while nylon-pad/sol-gel specimens exhibit ~85% cohesive failure. Once again, the data is very repeatable, with the three sets of data are grouped together in Figure 3 versus randomly scattered.

Since the repeatability assessment was performed on Al 7075-T6 with 3M medium Roloc™ pads instead of Al 2024-T3 with 3M coarse Roloc™ pads as in Section 2.2.2, a second set of data were generated comparing the difference in strengths or failure modes due to different abrasive pads. The main concern was the difference in nylon-pad/sol-gel wedge test failure modes from Table 8 (95% cohesive failure on Al 2024-T3) compared to nylon-pad/sol-gel wedge test failure modes in Figure 3 (85% cohesive failure on Al 7075-T6). Results from Figure 4 show that both medium and coarse Roloc™ pads provide cohesive failure modes when tested in lap shear at ambient-temperature. When testing lap shear strength at 160°F, the coarse pads appear to provide slightly better failure modes than the medium pads while specimens yielded very similar strengths regardless of the failure mode, as shown in Figure 5. Finally, wedge tests show the coarse pads provide slightly better failure modes than the medium pads Figure 6.

4 CONCLUSIONS

Ambient-temperature adhesive bonding processes were investigated for on-aircraft repair of aluminum alloys in this program conducted by UDRI and AFRL. These process evaluations included both adhesives and prebond surface preparations. The two-part epoxy paste adhesives evaluated were Hysol products EA 9309.3NA, EA 9320NA, EA 9330.3, EA 9394, and EA 9396 as well as 3M Company EC 2615 and EC 3333. These adhesives exhibited a wide range of lap shear and peel properties. They were tested with three surface preparations: PAA/BR 127, nylon-pad/sol-gel and scuff-sand/solvent wipe. The latter two are ambient-temperature processes that do not include an adhesive primer. Lap shear, peel, and wedge tests were used to examine adhesive performance with these surface preparations.

Testing with the different surface preparations provided valuable information concerning bonded repairs. The scuff-sand/solvent wipe process, often used for on-aircraft repairs, not only resulted in decreased moisture durability but also reduced initial bond strengths. The nylon-pad/sol-gel process always outperformed scuff-sand/solvent wipe. This sol-gel procedure is nearly as simple to perform as scuff-sand/solvent wipe and does not contain hazardous materials other than the solvent found in both processes. The nylon-pad/sol-gel process consisted of a solvent wipe using

acetone and abrasion with 3M medium Roloc™ pads followed by blowing off excess debris using compressed dry, clean, nitrogen. Clean, filtered air could be used in lieu of nitrogen. After debris removal, sol-gel solution was applied so surfaces were kept wet for 3 minutes, then adherends were dried at ambient temperature (70°F) for 30 minutes prior to adhesive application.

The PAA/BR 127 process produced the best overall results as was expected since it is the premier aluminum surface preparation. It provided excellent initial bond strengths and long-term resistance to moisture degradation. However, in this study, EA 9320NA adhesive used in conjunction with the nylon-pad/sol-gel surface preparation in an ambient-temperature process provided results nearly as good as those obtained using PAA/BR 127 with the same adhesive. The other Hysol adhesives did not provide the same level of performance as EA 9320NA when used in conjunction with the nylon-pad/sol-gel surface preparation. The 3M adhesives exhibited gross amounts of interfacial failure with all surface preparations evaluated in this program.

Specimens prepared with the nylon-pad/sol-gel surface preparation and bonded with EA 9320NA proved to provide repeatable results. Good results were achieved on both AL 2024-T3 and AL 7075-T6 using both 3M medium and coarse Roloc™ pads. However, further testing proved that slightly better failure modes were achieved when abrading with 3M coarse Roloc™ pads.

For the best strength and moisture durability results without the use of acids, grit-blast/sol-gel or grit-blast/silane processes should be used. Though not as convenient as nylon-pad/sol-gel, these surface preparations are now utilized for on-aircraft bonding. The nylon-pad/sol gel process should be considered for applications that currently employ scuff-sand/solvent wipe. Without primer, the nylon-pad/sol-gel surface preparation provides better bond strength and durability than scuff-sand/solvent wipe, yet it remains a simple, quick, and practical on-aircraft process. The use of adhesive bond primer with nylon-pad/sol-gel increases bond durability[14], but it is it no longer an ambient-temperature process.

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